

# X-ray powder diffraction data for the mineral refikite

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X-ray powder diffraction data, unit-cell parameters and space group for a rare natural organic mineral refikite, C<sub>20</sub>H<sub>32</sub>O<sub>2</sub>, abiet-13(15)-en-18-oic acid, are reported [ $a = 22.55(2) \text{ \AA}$ ,  $b = 10.469(6) \text{ \AA}$ ,  $c = 7.930(9) \text{ \AA}$ , unit-cell volume  $V = 1871.82 \text{ \AA}^3$ ,  $Z = 4$  and space group  $P2_12_12$ ]. All measured lines were indexed and are consistent with the  $P2_12_12$  space group. No detectable impurities were observed. © 2012 International Centre for Diffraction Data. [doi:10.1017/S088571561200053X]

Key words: X-ray powder diffraction, mineral refikite, abiet-13(15)-en-18-oic acid, 1, 4a-dimethyl-7-(1-methylethylidene)-tetradecahydrophenanthrene-1-carboxylic acid

## I. INTRODUCTION

The organic mineral refikite belongs to a rare group of natural hydrocarbons containing oxygen such as flagstaffite, hoelite and guanine, and was first described by Strunz and Contag (1965) from Kolbermohr, Bavaria, Germany, as needle-like crystals in roots of fossil spruce in a swamp. Refikite, summary formula C<sub>20</sub>H<sub>32</sub>O<sub>2</sub> or C<sub>19</sub>H<sub>31</sub>COOH, trivial name abiet-13(15)-en-18-oic acid, systematic name 1,4a-dimethyl-7-(1-methylethylidene)-tetradecahydrophenanthrene-1-carboxylic acid, belongs to abietic-type resin acids (Figure 1). The systematic name differs from the one given by Strunz and Contag (1965) and was established on the basis of single-crystal structure of the mineral by the first author of this article.

The original description of mineral (Strunz and Contag, 1965) included the correct determination of non-centrosymmetric space group  $P2_12_12$  from Weissenberg photographs, which was confirmed by single-crystal measurement.

## II. EXPERIMENTAL

### A. Sample preparation

The sample is formed by white to white-yellowish polycrystalline crusts and occasional transparent rare crystals growing on pine tree bark. The maximum size of crystals is about 1 mm, the coatings take several square centimetres. The samples were found at the locality V Borkách, near Krásno nad Teplou, Slavkovský les Mountains, western Bohemia, Czech Republic, Central Europe, under bark of peated wood. The natural sample was characterized by powder and single-crystal diffraction, as well as by nuclear magnetic resonance (NMR) and HR MS (negative electrospray,  $[M-H]^- = 305.2481 \text{ Da}$ ).

### B. Diffraction data collection and reduction

The diffraction pattern for the title compound was collected at room temperature using an X'Pert PRO  $\theta$ - $\theta$  powder

diffractometer with parafocusing Bragg-Brentano geometry and CuK $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ , generator setting: 40 kV, 30 mA). Owing to a small amount of the sample, it was placed on the surface of a silicon zero-background sample holder. To gain intensities, the Soller slits used for the measurement were 0.04 rad instead of 0.02 rad used for collecting a standard ICDD pattern that results in slightly higher background intensities level.

An ultrafast X'Celerator detector was employed to collect XRD data over the angular range from 7° to 70°  $2\theta$  with a step size of 0.017°  $2\theta$  and a counting time of 40.64 s per step. Data evaluation was performed using the software package HIGHSCORE PLUS V 3.0c PANalytical, Almelo, Netherlands.

Automatic indexing of the experimental XRD pattern was done using TREOR (Werner *et al.*, 1985). Space group of the original card 00-028-2009 was established from Weissenberg photographs (Strunz and Contag, 1965) and was verified by single-crystal measurement.

## III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2 up to 40°  $2\theta$  as no diffraction lines were observed at higher angles. Automatic indexing results obtained by TREOR show that the title compound is orthorhombic with space group  $P2_12_12$  and unit-cell parameters:  $a = 22.55(2) \text{ \AA}$ ,  $b = 10.469(6) \text{ \AA}$ ,  $c = 7.930(9) \text{ \AA}$ , unit-cell volume  $V = 1871.82 \text{ \AA}^3$  and  $Z = 4$ . The figures of merit are  $F_{19} = 9$  (0.020001, 116) (Smith and Snyder, 1979) and  $M_{19} = 6$

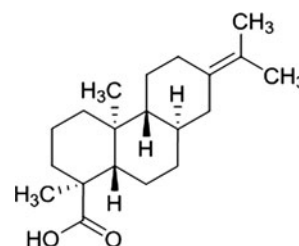


Figure 1. Structural formula of the organic mineral refikite, abiet-13(15)-en-18-oic acid.

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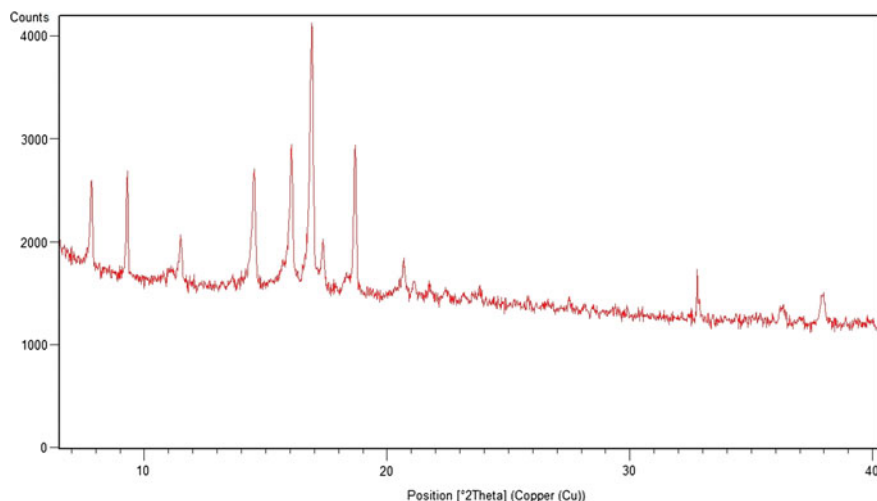


Figure 2. X-ray powder diffraction pattern of the mineral refikite using  $\text{CuK}\alpha_1$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ).

TABLE I. Indexed X-ray powder diffraction data for the organic mineral refikite,  $\text{C}_{20}\text{H}_{32}\text{O}_2$ , abiet-13(15)-en-18-oic acid, are reported [ $a = 22.55(2) \text{ \AA}$ ,  $b = 10.469(6) \text{ \AA}$ ,  $c = 7.930(9) \text{ \AA}$ , unit-cell volume  $V = 1871.82 \text{ \AA}^3$ ,  $Z = 4$  and space group  $P2_12_12_1$ ]. All measured lines were indexed and are consistent with the  $P2_12_12_1$  space group.

$2\theta_{\text{obs}}$ ( $^\circ$ )	$d_{\text{obs}}$ ( $\text{\AA}$ )	$I_{\text{obs}}$	$h$	$k$	$l$	$2\theta_{\text{calc}}$ ( $^\circ$ )	$d_{\text{calc}}$ ( $\text{\AA}$ )	$\Delta 2\theta$
7.825	11.289	34	2	0	0	7.836	11.274	0.0104
9.297	9.505	40	1	1	0	9.307	9.495	0.0096
11.494	7.692	18	2	1	0	11.526	7.671	0.0315
14.518	6.096	43	3	1	0	14.496	6.105	-0.0216
16.062	5.514	55	2	1	1	16.062	5.514	0.0002
16.902	5.242	100	0	2	0	16.925	5.234	0.0235
17.355	5.106	21	1	2	0	17.379	5.099	0.0235
18.329	4.836	9	3	1	1	18.324	4.838	-0.0050
18.684	4.745	57	2	2	0	18.675	4.748	-0.0086
20.682	4.291	15	1	2	1	20.694	4.289	0.0126
21.107	4.206	8	4	1	1	21.100	4.207	-0.0070
21.746	4.084	8	2	2	1	21.801	4.073	0.0549
23.806	3.735	9	2	0	2	23.769	3.740	-0.0372
27.502	3.241	6	4	0	2	27.481	3.243	-0.0214
32.775	2.730	20	6	0	2	32.809	2.728	0.0338
36.182	2.481	9	8	2	0	36.168	2.482	-0.0140
36.328	2.471	10	1	4	1	36.338	2.470	0.0094
37.897	2.372	13	4	4	0	37.871	2.374	-0.0264
37.975	2.368	13	7	3	0	37.986	2.367	0.0110

(de Wolff, 1968). All lines were indexed and are consistent with the  $P2_12_12_1$  space group.

Considering the quality of the natural organic sample and the scarcity of the mineral, the presented data of 19 diffraction lines (Table I) represent an improvement over the current card 00-028-200 that contains seven diffraction lines.

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